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Semiannual Report No. 2

Contract AF 33(657)-7590

A Study for Materials for IR Detectors

January 20, 1963

SYRACUSE UNIVERSITY RESEARCH INSTITUTE

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Introduction

Work on this contract was started in February 1962, with the aim of finding new materials useful as IR detectors. Several possibilities have been investigated to date. The material In-Ga-Sb with various proportions of In and Ga has shown most promise and the progress on this work is described on the following pages.

The work described here was performed by Mr. Joseph Wrobel assisted by Mr. Frank Renda. Dr. Henry Levinstein is project director.

It was shown in report No. 1 that solutions of InSb and GaSb have a long wavelength absorption threshold which depends on the relative proportions of these compounds. During the last period an investigation of photoconductivity in the system In-Ga-Sb has begun in the hope that similar behavior would be observed.

Homogeneous ingots of the polycrystalline alloy were obtained by mixing pure InSb and GaSb in the ratio of 7:1; this mixture is then quenched and subsequently zone leveled at a rate of 1/2" per day. Usually the first 1/2" of the ingot is inhomogeneous due to a rapid freeze out caused by a supercooling of the melt. A seed of GaSb provides nucleation sites for the melt, thereby reducing the rapid freeze out, and ingots which are completely homogeneous have been obtained. It was hoped that the seed would also produce single crystal ingots. However, all ingots obtained so far have been polycrystalline.

Samples for photoconductivity were cut to a size of 3 x 3 x 3 mm from various regions of an ingot. The cubes were lapped with 900 mesh carborundum to provide a uniform surface for etching, and then etched in a mixture of nitric, hydrofluric acid, and doubly distilled water. Due to preferential etching along grain boundaries, the sample can be etched for only a short time. Otherwise, deep etch pits are formed which penetrate into the bulk of the sample thereby resulting in excessive noise of the photoconductor, and increasing the tendency for the sample to break when handled. The samples were mounted on 10-32 screws using indium solder. The screws in turn were attached to the sample holder. The screw makes possible the rapid exchange of samples. Preliminary measurements to observe whether photoconductivity exists were made by mounting the samples in a "dip tube", a hollow monel metal

metal tube (Fig. 1). The sample is mounted at one end, a blackbody and chopper at the other. Leads are placed in an insulated hollow tube soldered on the outside of the "dip tube". Measurements of photoconductive signal can thus be made while the sample is immersed in the coolant. The device provides a means for evaluating many . samples in a short time with little waste of liquid helium. Those samples which showed large photoconductive signals were then mounted in a conventional dewar for more careful evaluation. In general, the samples were first immersed in liquid nitrogen, then in liquid helium. In all cases sample resistances in liquid nitrogen were of the order of 10 ohms and no signal was observed. When placed in the helium storage tank, sample resistance rose to as much as 100,000 ohms. Photoconductivity with signal to noise ratios of 30:1 → 150:1 was observed. The samples with the greatest signal to noise ratios were mounted in a Hoffmann helium dewar and spectral response measurements were made on a Perkin-Elmer Model 13 monochromator. Fig. 2 shows the spectral response curves of several samples with different composition. It should be noted that by varying the composition of the alloy, different cutoff energies were obtained, with cutoffs similar to those observed in absorption.

While the photosensitivity obtained in the 3 - 5 μ region is encouraging, no useful detector can be prepared until a similar response may be obtained with considerably less cooling. Since liquid helium cooling was required for a material with so large an energy gap, it was assumed that a shallow impurity produced a large number of charge carriers which were only frozen out at liquid helium temperature. Resistance and Hall constant vs temperature was measured on

several samples. These measurements indicate an impurity level with an activation energy of .01 ev (Fig. 3), that the impurities are "p" type with a concentration of about 2 x 10^{15}/cc. In order to obtain photoconductivity at higher temperatures, it is necessary to remove this impurity. The impurity appears to be introduced by the GaSb. All attempts to purify the alloy and also GaSb by multiple zone refining proved fruitless. The alternative to purification would be the introduction of donor impurities into the ingot in order to compensate this level. Several ingots were doped with tin which is usually "n" type in InSb. However, the alloy became increasingly "p" type. Thus tin is not a suitable compensating impurity. Se is "n" type in both InSb and GaSb, hence one would expect it to be "n" type in the alloy. An ingot doped with Se was grown, and Hall measurements indicated that a region of conversion from "p" to "n" type existed in the ingot. A sample cut from this region and mounted in a liquid nitrogen dewar had a sample resistance at litrogen temperature of 4000 ohms and showed a photosignal. Fig. 4 shows the relative spectral response of the sample.

Se as a dopant to compensate the alloy, has the disadvantage of a high vapor pressure. Because of this Se will tend to boil out of the zone and control is difficult. It is therefore necessary to search for an impurity which is "n" type in the alloy, and has a low vapor pressure so that exact compensation of the .01 impurity level can be attained. In this way, it should be possible to construct a detector operating at room temperature.

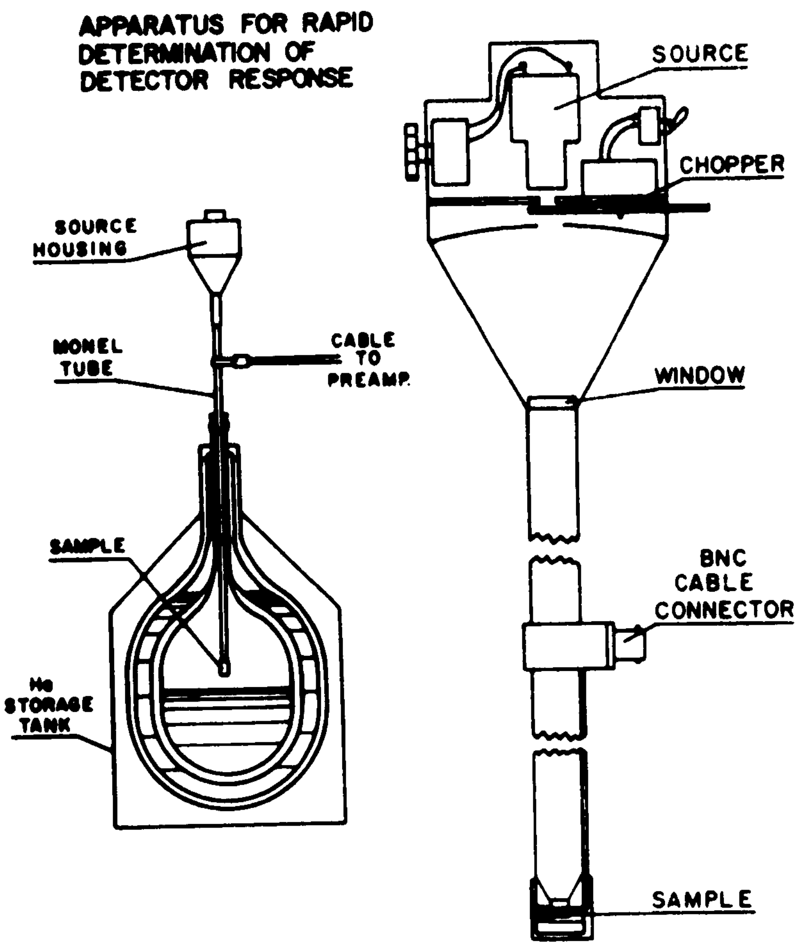

APPARATUS FOR RAPID
DETERMINATION OF
DETECTOR RESPONSE
SOURCE HOUSING
MONEL TUBE
CABLE TO PREAMP.
SAMPLE
He STORAGE TANK
SOURCE
CHOPPER
WINDOW
BNC CABLE CONNECTOR
SAMPLE

FIG. 1

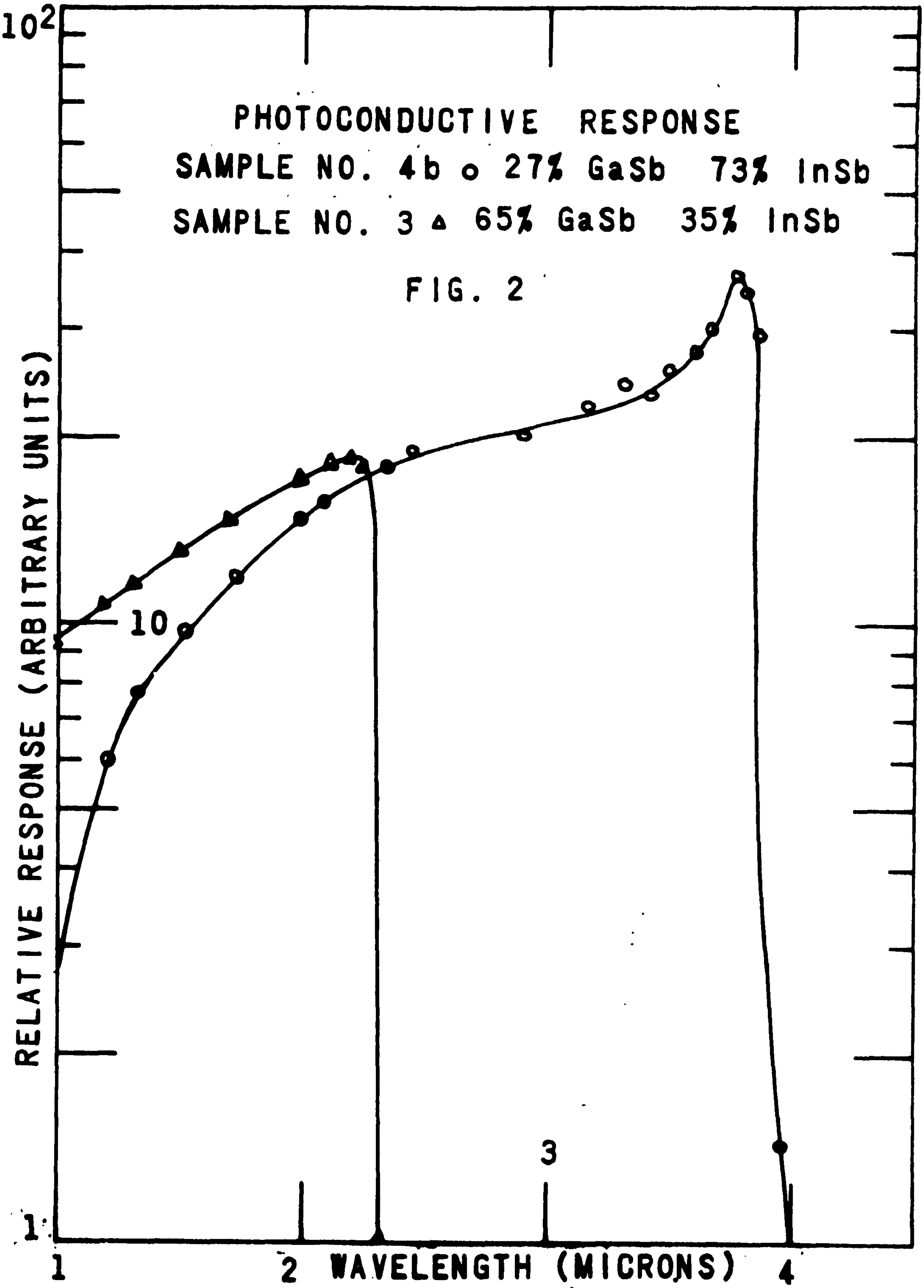
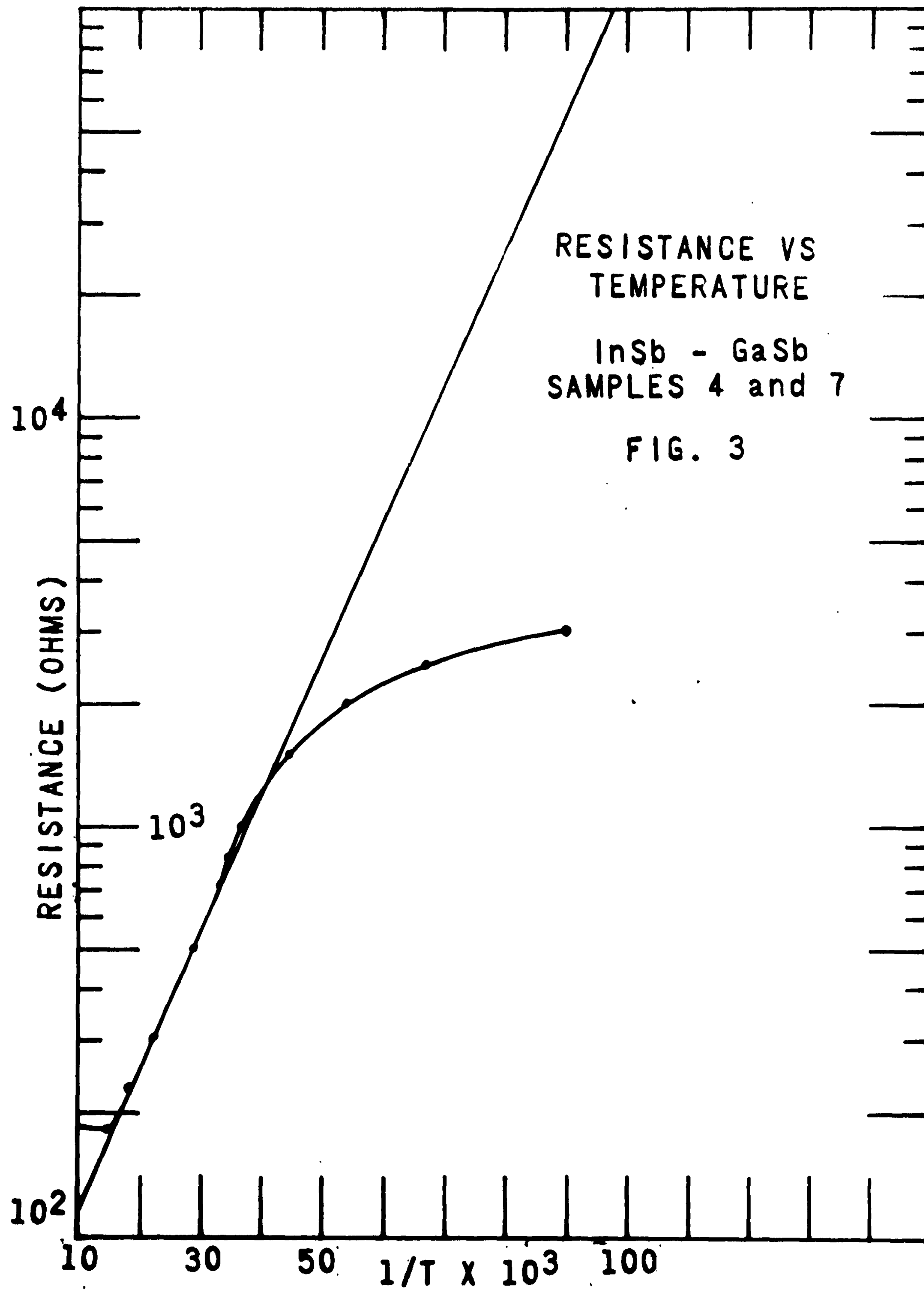

PHOTOCONDUCTIVE RESPONSE
SAMPLE NO. 4b o 27% GaSb 73% InSb
SAMPLE NO. 3 △ 65% GaSb 35% InSb
RELATIVE RESPONSE (ARBITRARY UNITS)
WAVELENGTH (MICRONS)
10^2
10
1
1
2
3
4

FIG. 2



RESISTANCE VS
TEMPERATURE
InSb - GaSb
SAMPLES 4 and 7
RESISTANCE (OHMS)
10^4
10^3
10^2
10
30
50
100
$1/T \times 10^3$

FIG. 3

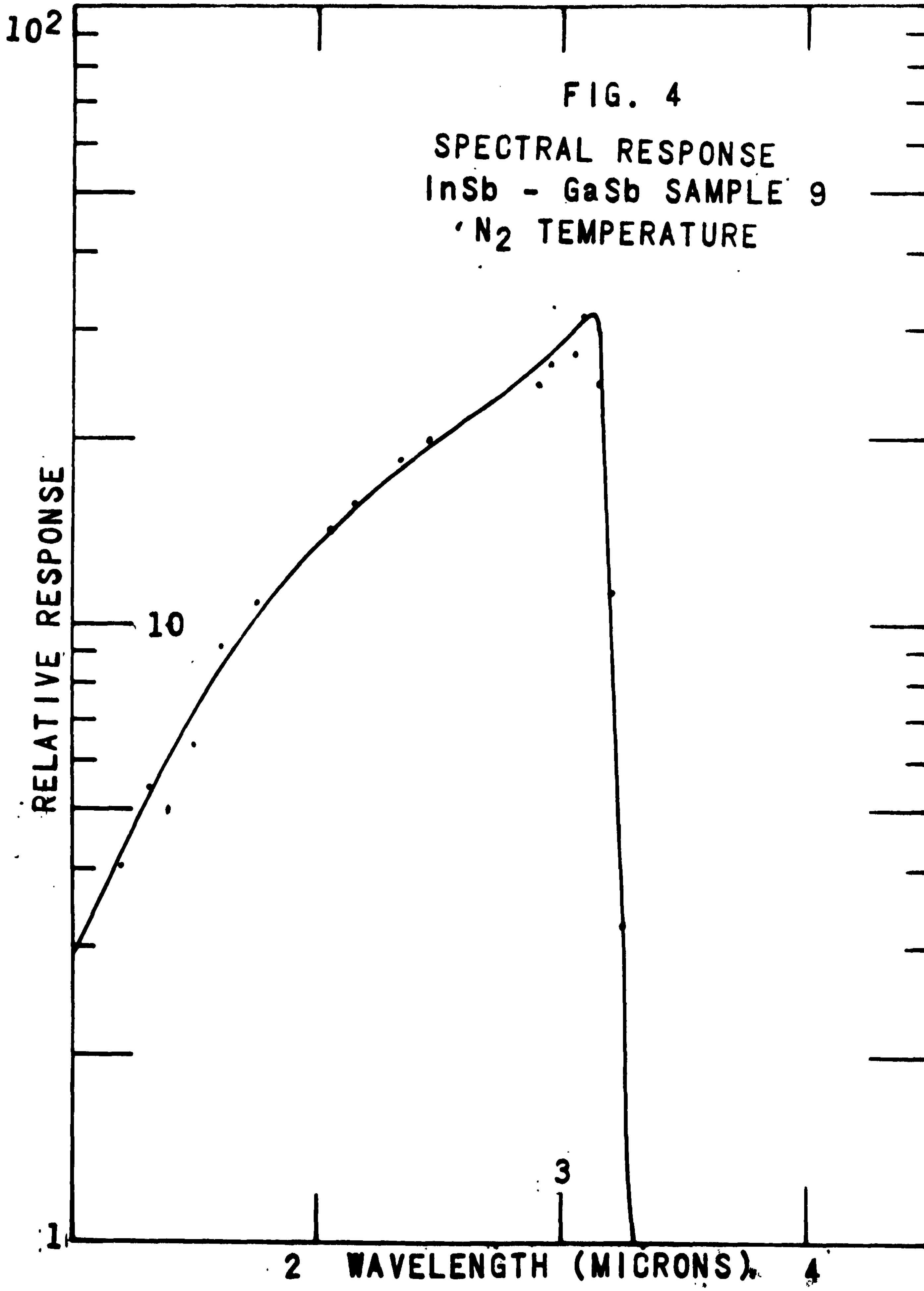


FIG. 4
SPECTRAL RESPONSE
InSb - GaSb SAMPLE 9
N_2 TEMPERATURE
10^2
10
1
RELATIVE RESPONSE
2
3
4
WAVELENGTH (MICRONS)

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